

EFFECT OF AGING ON CORROSION BEHAVIOUR OF AA6061 ALUMINIUM
ALLOY

MOHD SYAHIDAN BIN MOHAMED NAWI
MA08128

Report submitted in partial fulfilment of the requirements for the award of Bachelor
of Mechanical Engineering

Faculty of Mechanical Engineering
UNIVERSITY MALAYSIA PAHANG

JUNE 2012

ABSTRACT

The material for experiments was an extruded sheet AA6061 aluminium alloys by thickness of 2 mm. We studied the influence of different artificial aging parameters on corrosion behaviour of Al-Mg-Si alloy. The Al alloys was solution treated at $490\pm 5^{\circ}\text{C}$ for 5 hours, quenched in oil at room temperature and artificial aging at 170°C , 190°C at different aging time of 60, 180 and 360 minutes respectively. After heat treatment process, the obtained alloys will be etched for microstructure seeking purpose and then were corroded in solution of 3.5% NaCl by conducting potentiodynamic polarization for electrochemical measurement. After corrosion test, samples were prepared for analyzing the surface morphology of corrosion formed after exposed to the chloride media. We observed that the lowest corrosion rate has the sample aged at 170°C for 1 hour. The highest corrosion rate happens at 190°C for 1 hour. At 190°C for 6 hours the maximum hardness is obtained, while corrosion behaviour is better for all samples by comparing with as-received sample.

ABSTRAK

Bahan yang digunakan untuk eksperimen ini adalah AA6061 aloi aluminium yang dihasilkan melalui proses penyemperitan dengan ketebalan 2 mm. Kajian yang dijalankan adalah berdasarkan pengaruh parameter penuaan tiruan yang berbeza ke atas kadar pengaratan aloi Al-Mg-Si. Aloi ini telah terawat haba pada suhu $490 \pm 5^{\circ}\text{C}$ selama 5 jam, dan disejukkan dengan cepat dalam minyak pada suhu bilik dan seterusnya proses penuaan tiruan pada 170°C , 190°C pada masa yang berbeza iaitu 60, 180 dan 360 minit masing-masing. Aloi terawat haba yang diperolehi akan dipunar dengan asid untuk mendapatkan struktur mikro aluminium aloi terawat haba dan seterusnya dijalankan ujian pengaratan di dalam larutan 35% NaCl melalui ujian pembelauan potentiodynamik untuk ukuran elektrokimia. Selepas ujian pengaratan, analisis morfologi terhadap permukaan sampel dijalankan untuk mengesan bentuk pengaratan yang terhasil selepas terdedah kepada klorida. Dari pemerhatian, kadar pengaratan terendah berlaku pada sampel yang melalui penuaan tiruan pada suhu 170°C selama 1 jam. Kadar pengaratan tertinggi yang berlaku adalah penuaan tiruan pada suhu 190°C selama 1 jam. Pada suhu 190°C selama 6 jam kekerasan maksimum diperolehi, manakala kelakuan pengaratan adalah lebih baik bagi semua sampel mengikut perbandingan dengan sampel kawalan.

TABLE OF CONTENTS

	Page
SUPERVISOR’S DECLARATION	ii
EXAMINER ’S DECLARATION	iii
STUDENT’S DECLARATION	iv
DEDICATION	v
ACKNOWLEDGEMENTS	vi
ABSTRACT	vii
ABSTRAK	viii
TABLE OF CONTENTS	ix
LIST OF TABLES	xiii
LIST OF FIGURES	xiv
LIST OF SYMBOLS	xvii
LIST OF ABBREVIATIONS	xix
 CHAPTER 1 INTRODUCTION	
1.1 The Objectives of Project	1
1.2 Problem Statement	2
1.3 Objectives	3
1.4 Project Scopes	3
1.5 Overview of the Report	4

CHAPTER 2 LITERATURE REVIEW

2.1	Aluminium Alloy	5
2.2	AA 6061 Aluminium Alloy	6
2.3	Precipitation Hardening	7
2.4	Solution heat treatment	7
2.5	Aging	8
2.6	Forms of Corrosion	9
	2.6.1 General corrosion	9
	2.6.2 Localized corrosion	9
2.7	Passivity of Aluminium Alloys	10
2.8	Corrosion Mechanisms	12
2.9	Corrosion Rates Measurement	13
	2.9.1 Electrochemical polarization	13
	2.9.2 Tafel extrapolation	14
2.10	Metallographic	15
	2.10.1 Metallurgical microscope	15
	2.10.2 Scanning electron microscope	16
2.11	Vickers Hardness Test	16
2.12	Conclusion	18

CHAPTER 3 METHODOLOGY

3.1	Introduction	19
3.2	Methodology Flow chart	20
3.3	Sample Preparation	22
	3.3.1 Solution heat treated	23
	3.3.2 Aging	23
	3.3.3 Surface analysis	26
3.4	Electrochemical Test	27
	3.4.1 Solution preparation	29
	3.4.1.1 Procedure for NaCl solution	30
3.5	Microstructural Examination	30
3.6	Performing Hardness Test	31
3.7	Analysis of Data	32
3.8	Conclusion	32

CHAPTER 4 RESULTS AND DISCUSSION

4.1	Introduction	33
4.2	Surface Analysis	33
4.3	Hardness	41
4.4	Potentiodynamic Polarization	43
	4.4.1 Corrosion rate	51
4.5	Pitting Mechanism	53

CHAPTER 5 CONCLUSION AND RECOMMENDATIONS

5.1	Introduction	55
5.2	Conclusion	55
5.3	Recommendations	56

REFERENCES	57
-------------------	-----------

APPENDICES

A	Specimen for electrochemical cell	59
B	Tafel extrapolation using IVMan Software	60
	Parameter needed to measure the corrosion rate using IVMan software	60
C	Gantt Chartt for FYP1 and FYP2	61

LIST OF TABLES

Table No.		Page
2.1	Wrought aluminium alloy groups	6
3.1	Sample preparation	22
3.2	Aging process	24
3.3	Composition of etchant for aluminium alloys	25
4.1	Sample preparation	41
4.2	Hardness values of as receive and the sample have undergone various aging time and temperature	41
4.3	Potentiodynamic setup parameter	43
4.4	Corrosion Rates Determined by Tafel Extrapolation Method in 3.5% NaCl solution	51

LIST OF FIGURES

Figure No.		Page
2.1	Quasi-binary phase diagram for Al-Mg-Si alloy indicating important transition zones	7
2.2	Localize corrosion of pitting form	10
2.3	Pourbaix diagram of aluminium	10
2.4	Polarization diagram	14
2.5	The indenter of Vickers hardness test	18
3.2	Sample dimension	22
3.3	Furnace	23
3.4 (i)	Sample preparation (a) cold mounting and embed the copper wire (b) grinding process	24
3.4 (ii)	Sample preparations polishing with 6 μ Polycrystalline diamond polishing with 3 μ Polycrystalline diamond and 1 μ Polycrystalline diamond	25
3.5	Etching process	26
3.6	Inverted optical microscope	27
3.7	Electrochemical cells interconnect with WPG 100 potentiostat and computer	28
3.8	Electrochemical cell	29
3.9	Optical measurement	30
3.10	Hardness test device	31
4.1	Microstructure as-receive aluminium; (a) At magnification 200x (b) At magnification 500x	34
4.2	Microstructure of aluminium after Solution treated at $490 \pm 5^{\circ}\text{C}$ for 5 hours and quenched in oil at room temperature followed by aging at 170°C for an hour; (a) At magnification 200x (b) At magnification 500x	35

4.3	Microstructure of aluminium after Solution treated at $490 \pm 5^{\circ}\text{C}$ for 5 hours and quenched in oil at room temperature followed by aging at 170°C for 3 hours; (a) At magnification 200x (b) At magnification 500x	36
4.4	Microstructure of aluminium after Solution treated at $490 \pm 5^{\circ}\text{C}$ for 5 hours and quenched in oil at room temperature followed by aging at 170°C for 6 hours; (a) At magnification 200x (b) At magnification 500x	37
4.5	Microstructure of aluminium after Solution treated at $490 \pm 5^{\circ}\text{C}$ for 5 hours and quenched in oil at room temperature followed by aging at 190°C for an hour; at (a) At magnification 200x (b) At magnification 500x	38
4.6	Microstructure of aluminium after Solution treated at $490 \pm 5^{\circ}\text{C}$ for 5 hours and quenched in oil at room temperature followed by aging at 190°C for 3 hours; (a) At magnification 200x (b) At magnification 500x	39
4.7	Microstructure of aluminium after Solution treated at $490 \pm 5^{\circ}\text{C}$ for 5 hours and quenched in oil at room temperature followed by aging at 190°C for 6 hours; (a) At magnification 200x (b) At magnification 500x	40
4.8	Hardness value of as-receive and heat treated sample at temperature of 170°C	42
4.9	Hardness value of as-receive and heat treated sample at temperature of 190°C	42
4.10	Experiment obtained in 3.5% NaCl solution for as-receive sample of aluminium alloy AA 6061 undergone (a) Potentiodynamic polarization (b) Tafel extrapolation plot	44
4.11	Experiment obtained in 3.5% NaCl solutions after solution treated at $490 \pm 5^{\circ}\text{C}$ for 5 hours, quenched in oil at room temperature followed by aging at 170°C for an hour undergone (a) Potentiodynamic polarization (b) Tafel extrapolation plot	45
4.12	Experiment obtained in 3.5% NaCl solutions after solution treated at $490 \pm 5^{\circ}\text{C}$ for 5 hours, quenched in oil at room temperature followed by aging at 170°C for three hours undergone (a) Potentiodynamic polarization (b) Tafel extrapolation plot	46

4.13	Experiment obtained in 3.5% NaCl solutions after solution treated at $490\pm 5^{\circ}\text{C}$ for 5 hours, quenched in oil at room temperature followed by aging at 170°C for six hours undergone (a) Potentiodynamic polarization (b) Tafel extrapolation plot	47
4.14	Experiment obtained in 3.5% NaCl solutions after solution treated at $490\pm 5^{\circ}\text{C}$ for 5 hours, quenched in oil at room temperature followed by aging at 190°C for an hour undergone (a) Potentiodynamic polarization (b) Tafel extrapolation plot	48
4.15	Experiment obtained in 3.5% NaCl solutions after solution treated at $490\pm 5^{\circ}\text{C}$ for 5 hours, quenched in oil at room temperature followed by aging at 190°C for an three undergone (a) Potentiodynamic polarization (b) Tafel extrapolation plot	49
4.16	Experiment obtained in 3.5% NaCl solutions after solution treated at $490\pm 5^{\circ}\text{C}$ for 5 hours, quenched in oil at room temperature followed by aging at 190°C for six hours undergone (a) Potentiodynamic polarization (b) Tafel extrapolation plot	50
4.17	The value of corrosion rate of as receive and the sample that have undergone various aging time and temperature	51
4.18	Surface morphology of AA6061 aluminium alloy after electrochemical test in the solution of 3.5% NaCl	53
6.1	Sample for electrochemical cell	59
6.2	Tafel extrapolation using IVMan Software	60
6.3	Parameters needed to measure the corrosion rate using IVMan software	60

LIST OF SYMBOL

A	Area
Al	Aluminium
A_a	Anode area
A_c	Cathode area
Al^{3+}	Aluminium dissolve to 3 electron
Al_2O_3	Aluminium oxide
$Al(OH)_3$	Aluminium hydroxide
$AlCl_3$	Aluminium chloride
$Al(OH)_2Cl$	Aluminium oxychlorides
e	electron
E	Potential
E_{corr}	Corrosion potential
H_2	Hydrogen gas
H^+	Hydrogen ion
HNO_3	Nitric acid
i_{corr}	Corrosion current density
i_a	Anode current
i_c	Cathode current
$I_{appl reversed}$	Reverse current applied
M	Metal
n	No. of positive ion and electron
Si	Silicon
ϕ_A	Anode potential

ϕ_c	Cathode potential
ϕ_{corr}	Corrosion potential
ζ	Polarization
β_a	Anodic Tafel slopes
β_c	Cathodic Tafel slopes
$^{\circ}\text{C}$	Degree Celcius

LIST OF ABBREVIATIONS

AA	Aluminium Association
ACS	America Chemical Society
ASTM	American Society for Testing and Materials
FYP	Final Year Project
HV	Hardness Value of Vickers hardness test
mmpy	Millimeter per year
NaCl	Sodium chloride
SCE	Saturated Calomel Electrode

CHAPTER 1

INTRODUCTION

1.1 INTRODUCTION

The wide range use of aluminium and aluminium alloy in the transportation industry such as aircraft and automotive is based on fame mechanical characteristics of these alloys with regard to low specific weight and corrosion resistance (Enescu et al., 2010). Aluminium alloys are alloys in which the main element is aluminium itself. Further studies have been carried out on the 6xxx aluminium alloy because of their technological importance and exceptional increase in strength obtained by precipitation hardening. The other excellent characteristics of these alloys which are it can be shaped easily, low density, their good surface properties and good weld ability and take along with low price these make them commercially attractive.

Aluminium alloy is processed in very large at low cost for mostly building and architectural design works in most developing countries and it is thus quite understandable why attention is focus this series of alloys. For this project, it will focus on 6061 aluminium alloys. AA6061 is one of the most alloys widely used in 6000 series and well known due to versatile heat treatable alloy. It provide medium and high strength depend on the requirement of application.

Some aluminium alloy can be strengthening by conducting a heat treatment process. The purpose of this process is to alter the mechanical properties by increasing the value of their strength, hardness and also their corrosion resistance. The process applicable for 6061 alloy is precipitation strengthening which involve three basic steps. The solution heat treatment is the first step in the precipitation-

strengthening process. Then, the sample is rapidly cooled to a lower temperature usually water and finally follows by artificially aging. The temperature of aging is between 15 and 25 percent of the temperature different between room temperature and the solution heat-treatment temperature.

The salient features of aluminium alloy which can resist the corrosion have made these alloys very commercial for several of application. When speak of corrosion, usually we referring to the chemical attack process on metals. The AA6061 and AA6063 aluminium alloy is marine grade that can achieve high strength and great corrosion resistance. Meanwhile, the AA7075 are the aluminium that heavily use in aircraft industry. The AA7075 aluminium alloy may possess more strength that marine grade alloy has but is much more susceptible to corrosion. In other word, while the alloys have formidable performance in aircraft industry, it will perform poorly in marine applications.

Fundamentally, the aluminium is a very active metal where its nature to oxidize quickly. While a weakness for the most metal, actually this is the key for aluminium ability to resist corrosion. The present of oxygen in air, soil and water will react instantly to form aluminium oxide. The oxide layer is chemically bond to the surface of aluminium. Thus, the layer present will prevent the aluminium core for further reaction. It is different in steel corrosion which the oxide layer continuously puffs up and flakes off then exposing other surface to corrosion. The aluminium has excellent corrosion in wide range of water and soil condition because of tough oxide film form on the surface and hence providing an excellent corrosion protection except in several special cases.

1.2 PROBLEM STATEMENT

The use of aluminium alloy in variety of application is due to the superior characteristic belonging to aluminium itself. Therefore, too many researchers have been devoted to study the mechanical properties of these alloys such as strength, weld ability, formability as well as the ability to resist the corrosion. Generally, the investigation corrosion behaviour of aluminium is due to its important application in industry especially for the structure purpose. Thus, the effect of variation aging time

on corrosion behaviour of AA6061 aluminium alloy which initially has been heat treated to different temperature has been investigate in this project. The purpose is to investigate whether the corrosion rate of AA6061 aluminium alloy is being affected by the variation of aging time and temperature. Then the result is being compared to the previous experiment that has been conducted by other researchers.

1.3 OBJECTIVES

The objectives of the project that need to be achieved are:

1. To study the effect of aging on corrosion behaviour of AA6061 aluminium alloy.
2. To investigate the effect of variation aging time and temperature of heat treatment AA6061 aluminium alloy.

1.4 PROJECT SCOPES

The focus area will be done based on the following aspect:

- i) AA6061 aluminium alloy sample preparation.
- ii) Metallography to reveal the microstructure of the sample.
- iii) Perform the solution heat treated of aluminium alloys at 490° C for 30 minutes and quenched in water.
- iv) Artificial aging at 170° C and 190° C at different time 1 hours, 3 hours, and 6 hours before water quenching.
- v) Evaluate the corrosion rate by using electrochemical test based on weight loss method.
- vi) Surface analysis by using Optical Microscope.
- vii) Microstructures analysis of corrosion behaviour by using Scanning Electron Microscope (SEM).
- viii) Using Vickers hardness test to analyze the hardness of each specimen.

1.5 OVERVIEW OF THE REPORT

This project has been arranged in five chapters. The introduction has been written in this chapter. The chapter 2 will explain for the literature review. The methodology is being told in chapter 3 while the result of the experiment being discussed in chapter 4. The last chapter which is chapter 5 will be conclusion and recommendations for the entire project.

CHAPTER 2

LITERATURE REVIEW

2.1 ALUMINIUM ALLOY

Aluminium alloy are alloy which aluminium is the predominant metal. Typical alloying elements are copper, manganese, silicon, magnesium, and product. There are two types of aluminum product which are wrought and cast aluminum alloy. Cast aluminum alloy commonly used in widespread applications for structural component due to its excellent castability, corrosion resistance and particularly high strength to weight ratio in the heat treatment condition. However, the used of this cast alloy still a step backward on wide range uses of wrought aluminum alloy even though casting types provide more economical production method. This is partly because of cast aluminum alloy may contain defects such as porosity, oxides and other factors.

The most commonly used aluminum alloy designation in the United States is that of the Aluminium Association is the wrought aluminium alloy. The classification of wrought aluminium alloy is classified according to their major alloying element based on four digits numerical designation is shown in Table 2.1 (William and Javad, 2006)

Table 2.1: Wrought aluminium alloy groups

Type	series
Commercially pure aluminium (99% min)	1xxx
Copper	2xxx
Manganese	3xxx
Silicon containing alloy	4xxx
Magnesium	5xxx
Magnesium and Silicon containing alloy	6xxx
Zinc containing alloy	7xxx
Other elements containing alloy	8xxx
Unused series containing alloy	9xxx

Adapted from: Edward (2010)

2.2 AA6061 ALUMINIUM ALLOY

The AA6061 aluminium alloy is known to be age hardenable alloy which containing magnesium and silicon as the predominant alloying element. The magnesium silicide is the form of interest to be formed in these series of alloy. The 6061 Al-Mg-Si having balanced ratio of 1% magnesium and 0.6% silicon to form MgSi has set up as the standard for light weight, economical for general structure use. To achieve more strength, copper is added about 0.3% in the T6 temper compared to the copper free alloys with balanced composition of Mg and Si (Edward, 2010). The 6061 alloy largely found in the market due to its features such as good corrosion resistance, weldability, and attractive surface appearance render these alloy very useful for extruded product.

2.3 PRECIPITATION HARDENING

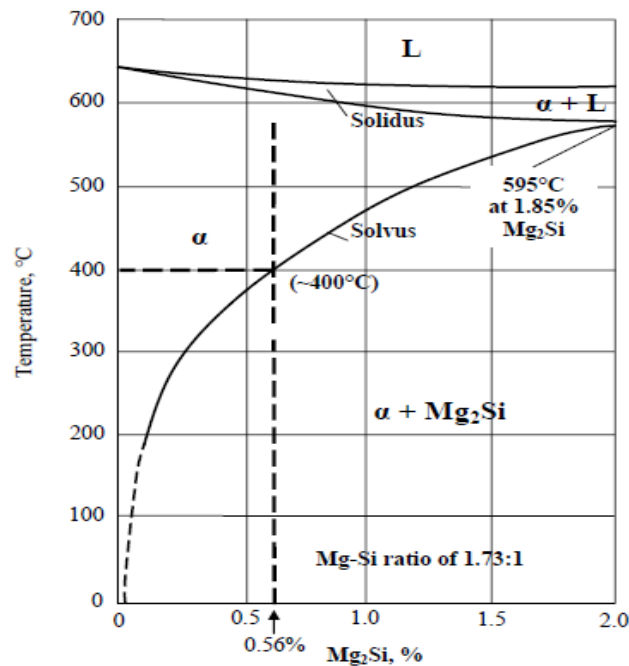


Figure 2.1: Quasi-binary phase diagram for Al-Mg-Si alloy indicating important transition zones

2.4 SOLUTION HEAT TREATMENT

The solution heat treatment or so called solution annealing is the first step to achieve precipitation hardening. The main purpose for this treatment is to put all the solute or second phase into solution. The alloy sample could be wrought or cast form is heated to a temperature lies midway between the solvus and solidus line. This process will cause single phase solid solution to form. While conducting the solutionizing, overheating and underheating should be avoid unless the desire properties such as tensile strength, fracture toughness and ductility will gradually decrease (Pat, 1999).

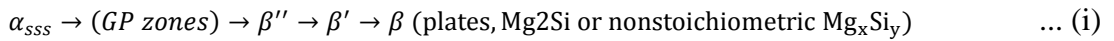
Quenching is a hardening heat treating and the quenching medium normally used is air, water, oil, or liquid polymers. As a result, supersaturated solid solution of second phase alloy will be form through this process.

2.5 AGING

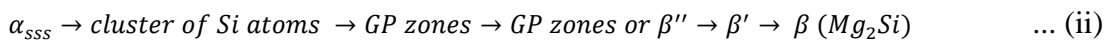
The aging hardening or precipitation hardening was first discovered in Germany when the hardness of aluminium-copper alloy was retested after it was laid in the laboratory for a week. The yielded hardness was much higher than before it was retested. Then the first name given is age hardening where the hardness gained as the alloy aged in time.

The purpose of precipitation hardening is to create a fine dispersion of precipitate particles. The particles then will resist the dislocation movement and hence strengthen the heat treated alloy. The alloy system itself should have the terminal solid solution where the solid solubility decreases with temperature to enable the precipitation hardening (William and Javad, 2006).

Due to the complex nature of precipitation, there are some difficulties to identify the chemical characteristics of fine scale microstructure. Consequently, the exact sequence of structural changes has faced controversy during aging. There are several precipitation sequences that have been proposed according to formation complexity. The general accepted sequence is:



Another sequence with more detail on the earlier stage of clustering and GP zone formation was proposed as:



The structure of alloy after water quenching will consist of supersaturated solid solution and can be noted as α_{ss} on the first stages of sequences above. When the supersaturated solid solution is heated at relatively low temperature, clusters of solute rich regions or Si are formed within the Al lattice and are completely coherent with it. These clusters are called GP (Guinier-Preston) Zones because they were first detected by Guinier and Preston. From the sequence, GP2 or β'' is where the peak hardness is achieved for wrought alloys. While β' is where the peak hardness is achieved for cast alloys. For β'' and β' , there is still confusion and unable to provide consistent evidence regarding the formation of both phases.

2.6 FORMS OF CORROSION

2.6.1 General Corrosion

The general corrosion is very regular form of corrosion. It can be uniform (even), quasi-uniform (near-uniform), or uneven. The term of general corrosion is referring on the greatest loss of metal or material. Oxidation, sulfidation, carburization, hydrogen effects, and also hot corrosion can be account as types of general corrosion.

However, general uniform corrosion is rare toward aluminium except in several special cases such as highly acidic or alkaline corrosive reagents. Aluminium alloys of the 1xxx, 3xxx, 5xxxx, and 6xxx series by many natural waters. Corrosions of aluminium is occurred when the presence of moisture and oxygen. The significant factors that involves in corrosion of aluminium include water pH, temperature, and conductivity. The conductivity is more toward the availability of cathodic reactant, presence of heavy metals, and corrosion potentials of the specific alloys. In chloride containing solutions, the corrosions occur regarding the pH range of 5.5-8.5 is less than either in distinctly acid or distinctly alkaline. In this pH range, aluminium is passive metal and normally undergoes localized corrosion rather than general uniform corrosion. However, the result obtained significantly related or depending on specific aluminium alloy under investigation.

2.6.2 Localized Corrosion

Localized corrosion is the most hazardous corrosion because it cannot be predicted easily just like general corrosion. There are several consequences affected by localized corrosion such as putting some equipment out of service and cause fatal accidents in a few circumstances.

Pitting type of corrosion usually concern in application involving passive metal and alloys in aggressive environment. Pitting corrosion of passive metal is commonly observed in presence of chlorides and other halides. Halide ions such as Cl^- can rise to severe localized corrosion. Generally, aluminium does not pit in

oxygen containing solution of nonhalide salts, because aluminium is not polarized to pitting potential at normal service. Pitting corrosion formed at weak points of the oxide or hydroxide passivating film of the alloy.

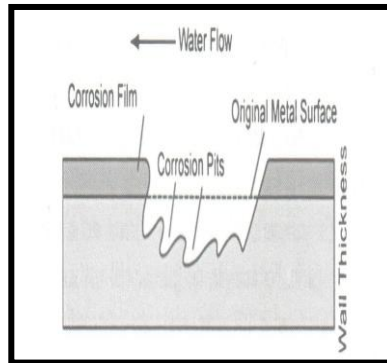


Figure 2.2: Localize corrosion of pitting form.

Source: Edward (2010)

2.7 PASSIVITY OF ALUMINIUM ALLOYS

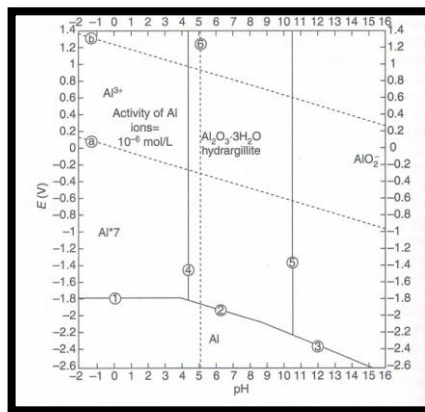


Figure 2.3: Pourbaix diagram of aluminium

Source: Edward (2010)

The role of thermodynamics has been used extensively to evaluate the corrosion tendency or pattern of metals. The figure above is describing the electrochemical potentials and equilibrium determined from the free enthalpy ΔG and the chemical equilibrium between the different metallic compounds in solution